INTERLABORATORY STUDY 91-4

OIL AND GREASE

(MISA TEST GROUP 25)

IN REAGENT WATER AND EFFLUENTS

MAY 1992

Ontario
Environment
Environnement

ISBN 0-7729-9696-2

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MAY 1992

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The author wishes to thank Sathi Selliah for the provision of the macros to perform the data evaluation process.

1 SUMMARY OF INTERLABORATORY STUDY 91-4

Interlaboratory Study 91-4 was initiated as part of an on-going program of laboratory performance management studies conducted by the Quality Management Office of Laboratory Services Branch, Environment Ontario. It assesses the performance of participating laboratories for the analysis of Oil and Grease (or Solvent Extractables) in spiked reagent water and in effluents. Forty-three laboratories (government, academic, industry, and commercial) agreed to participate in this study. Results were received from forty-one participants, with two participants also reporting duplicate results. The test is MISA Analytical Test Group 25[1].

The results demonstrate a low bias among the participating laboratories in this study. More consistent within-laboratory precision was observed for the participants using Dichloromethane as the extraction solvent, than by the participants using Freon as the extraction solvent. As well as the environmental concerns involved in the use of Freon and other Chlorofluorocarbons, this solvent does not appear to produce as consistent analytical performance as does the use of Dichloromethane. Variable performance was also observed for the analysis of the unspiked effluent samples, indicating the need for careful application of the method. There is also more variability in the results in this study, than in the previous MOE Interlaboratory Study involving Oil & Grease[5]. While there are more participants in this study than in the first study, it is not clear why consistent performance has not been maintained. A summary of the findings for the spiked reagent water samples is given below in Table I.

The low bias and the variability of results in this study stress the need for reference standards and matrix materials for this analytical procedure. It is hoped that laboratories will encourage the manufacture of these types of materials and continue to strive for consistent performance.

TABLE I
SUMMARY OF STUDY FINDINGS FOR SPIKED REAGENT WATER SAMPLES

Rank Number of Participants = 41	Rank vs Target	Rank vs Interlaboratory Median
Acceptable Performance	7 (17.1%)	7 (17.1%)
Erratic or Out of Control	13 (31.7%)	12 (29.3%)
Low Bias (Slope or Intercept)	20 (48.8%)	12 (29.3%)
High Bias (Slope or Intercept)	1 (2.4%)	10 (24.4%)

2 INTRODUCTION

Interlaboratory performance studies are conducted to assess the comparability and accuracy of data among different laboratories. These studies are useful for the identification of biases, precision and accuracy problems. Participation in such studies can serve as a guide for improving individual laboratory performance and maintaining performance standards. The Quality Management Office, Laboratory Services Branch, Environment Ontario has instituted an on-going program of interlaboratory studies to assess and enhance the performance of environmental laboratories providing analytical services.

This study was designed to assess the analytical variability among laboratories for the analysis of Oil and Grease or Solvent Extractables in spiked reagent water and in unspiked natural effluents. This test was chosen from the MISA (Municipal and Industrial Strategy for Abatement) General Regulation[1]. Participants were requested to use methods which conformed to the MISA analytical principles and protocols given in the General Regulation[1].

Forty-three laboratories (government, academic, industry, and commercial) agreed to participate in this study. Results were received from forty-one participants, with two participants reporting duplicate results. A list of participants is given in Appendix 2. Each participant was assigned a unique identification code to maintain confidentiality.

A set of five samples was distributed to each of the forty-three participants. The samples consisted of a reagent water blank and two reagent water samples fortified with motor oil, and two different unspiked effluent samples. Section 3 describes sample preparation, sample distribution, analytical methodology, and data evaluation procedures. Final results are tabled in Appendix 1 and discussed in Section 4.

3 PROCEDURE

3.1 Preparation of Samples

A stock spiking solution consisting of motor oil (10W30) was prepared in acetone and sealed into ampoules.

Samples were prepared in 1000 mL amber glass bottles. To each bottle, 1000 mL of distilled, deionized water was added by weight (1000 g ±2 g). It was assumed that the density of the water equalled 1.0. The unspiked samples were labelled D4.

The spiked samples were prepared by individually spiking the sample bottles with the appropriate amount of stock solution using the appropriate size of microlitre syringe. Target values are included in the tables in

Appendix 1. The Low Spike was labelled D5 and the High Spike was labelled D6.

Two different bulk effluent samples were used for this study. Each effluent was separately collected in a stainless steel tank and stirred overnight to form a homogenous mixture using a stirrer with a Teflon shaft and propeller. 1000 mL (1000 g ±2 g) of effluent was poured into amber glass bottles while continuously stirring the bulk sample. One effluent was from a sewage treatment plant and was labelled E4. The other effluent was combined from several different industrial and municipal sources and labelled E5. Any possible error introduced by the density of the effluent was considered too small to be significant.

3.2 Sample Distribution

Samples were packed and stored overnight at 4°C. Samples were shipped the following day by Purolator Courier to the participating laboratories. A list of the laboratories receiving sample sets is given in Appendix 2. Samples were shipped on October 29, 1991. A copy of all correspondence is also included in Appendix 2.

Several participants telephoned to report missing or broken samples on October 30. Replacement samples were provided to Laboratories 9019 and 9041. The QMO had insufficient extra samples to replace broken bottles for those participants who telephoned after October 30.

3.3 Analytical Methodology

Participating laboratories were requested to analyze the samples using routine in-house methods that complied with the principles and protocols outlined in Schedule 3 of the MISA General Regulation[1]. Participants were requested on the report form provided (Appendix 2) to summarize their Sample Preparation Principles, Instrumental Measurement Method Principles, and indicate the solvent used for the extraction. All participants were assigned a unique identification code.

3.4 Data Evaluation Technique

Results were submitted to the Quality Management Office, LSB in written form. All data were manually entered by laboratory code into an electronic spreadsheet.

The participating laboratories who reported results were mailed a copy of the tables of results on January 7, 1992. One participant reported a correction to their data. Both values are included in the tables, though the corrected result is used in the evaluation.

The mean, median and standard deviation were calculated for each parameter in each sample and are included in Table 3, Appendix 1. Results of the spiked reagent water samples were converted to percent recovery of the target value and are given in Table 2, Appendix 1.

The results were evaluated using the technique outlined in Reference 2. This Automated Evaluation Technique is summarized as follows:

1. Two samples at different concentrations are split among a number of laboratories and analyzed as requested. Results are entered into an electronic spreadsheet and programs (macros) are coded to perform the manipulations. The evaluation technique is performed after the participants have verified their results (see above).

2. High sample data evaluation:
   - i) Reject all results which differ from the median (H_m) by more than 10%.
   - ii) Calculate median (H), mean and standard deviation (S_h).
   - iii) Reinclude results if within 3 times S_h.
   - iv) Reiterate ii) and iii) until no further results are included.
   - v) Calculate relative standard deviation of final selected results (CV_h).

3. Low sample data evaluation:
   - i) Use 3 X CV_h X median(L_m) to exclude possible outliers.
   - ii) Calculate median (L), mean and standard deviation (S_l).
   - iii) Reinclude results if within 3 times S_l.
   - iv) Reiterate ii) and iii) until no further results are included.

4. Paired sample performance criteria:
   - i) Examine ratio of S_h/S_l: If <2, use results as reported in concentration units. Otherwise convert results to percent recovery based on target value, if known, or use recovery relative to median value - H, L.
   - ii) Prepare paired sample scatter diagrams of all results (Youden plots[3]).
   - iii) Calculate perpendicular distances from each point to the two 45° lines (Slope and Intercept error lines) and select the lesser of the two perpendicular distances (PD).
   - iv) Determine the median PD.
   - v) Determine the mean of all PD values less than 2.5 times the median and use this mean to estimate the repeatability S_w.
   - vi) Set warning limits for repeatability: 2 times S_w.
   - vii) Set control limits for repeatability: 3 times S_w.
   - viii) Set warning limits for possible bias: 3 times S_w (same as vii).
   - ix) Set control limits for possible bias: 4.5 times S_w.

5. Code performance and summarize in table
   i) In upper left or lower right quadrant - Erratic
   ii) In lower left or upper right quadrant - Biased Low or High
   iii) On horizontal or vertical axis - Out of Control
   iv) On diagonal line through origin - Slope or Standard problem
   v) On diagonal line not through origin - Intercept or blank problems.

For this study, the above evaluation technique was applied to the spiked reagent water samples (D5 and D6). The samples were not corrected for any background levels reported in the unspiked sample (D4), as most participants did not demonstrate any laboratory contamination. Some participants did appear to have some laboratory contamination, but their results were not handled in a different manner than those who did not report anything present in sample D4. No effect on the ranking would occur if the results were "blank corrected".

The spiked reagent water samples were evaluated both as a total group and then subdivided according to solvent used for the extraction procedure. Two different solvents were used by the majority of participants: Dichloromethane and Freon. Two participants used Petroleum Ether and one participant used Hexane. These three laboratories were included in the overall evaluation but were excluded from the separate evaluations of participants using Dichloromethane or Freon.

The initial evaluation of the spiked reagent water samples (D5 and D6) was done using the target values as the centre of reference. The diagrams are in Figures 1-3. Due to the low bias of the majority of the results, the results were re-evaluated using the interlaboratory median as the centre of reference. The diagrams are in Figures 4-6. The outcome of both evaluations are given in Table 4. The repeatability (Step 4 v) above) differs between the two evaluations, as the location of the error lines is shifted, based on the centre of reference. Both values of S_w are given in Table 3. The effect of the different evaluations based on the different centre of reference is discussed in Section 4.

The variability in the results for the effluent samples made them unsuitable for the Automated Evaluation Technique described above. The results are presented using a Youden Plot in Figure 7 and are discussed in Section 4.

4 DISCUSSION

OVERVIEW OF INTERLABORATORY PERFORMANCE

The overall results from this study show a low bias for the majority of participants compared to the target values. The interlaboratory mean and median for both

samples is lower than the target by approximately 20%. Only one participant had results that were biased high.

The majority of results are spread along the Concentration Dependant Error line (Figure 1 and 1A). For analyses that involve measurements against a standard, this type of distribution and low bias is usually attributable to differences in standards between the laboratories. However, the Oil & Grease or Solvent Extractables test is a general test that does not involve comparison of sample results against known standards. Low biases in this procedure may be attributed to improper timing of the steps in the procedure, poor solvent efficiency or problems with the evaporation steps (water bath or oven temperature too high)[4]. Rinsing of the sample bottle with an aliquot of the extraction solvent may also improve recovery, as material of interest may adhere to the sides of the bottle.

There were more participants with erratic or out of control performance than expected. The previous MOE Interlaboratory study involving this test[5] had fewer participants, but all the participants demonstrated reasonable within-laboratory precision in the first study. The between-laboratory standard deviation is higher in this study than the earlier study, reflecting the impact of the erratic participants. This implies inconsistency on the part of these participants in applying their analytical procedure.

The results were separated according to solvent used and evaluated separately as described in Section 3.4. There is no significant difference in the means or the variability between the two solvents (using the appropriate *t*-test and *F*-test). However, using the Automated Evaluation Technique[2], there is more variability in the Low Sample (D5) results by the participants using Freon than those using Dichloromethane. The Evaluation Procedure has produced a Selected Standard Deviation (Section 3.4, Step 2 ii-iv)) that suggests less variability of the High Sample (D6) by the participants using Freon than those using Dichloromethane (Appendix 1, Table 3). However it should be noted that the Selected Standard Deviation for Freon for Sample D6 is calculated from only 6 results, which is less than 50% of the reported values. This indicates that more than 50% of the results differ from the interlaboratory median by more than 3 times the Standard Deviation, indicating a high degree of between-laboratory variability. The repeatability also differs between the two solvents. The value for S_w for Dichloromethane is smaller than S_w for Freon (Appendix 1, Table 3). This indicates tighter within-laboratory control and results in a smaller area of acceptable performance. As a result, there are more laboratories using Freon that are flagged as Erratic or Out of Control as compared to those using Dichloromethane (Appendix 1, Table 4). 41% of the participants using Freon are flagged as Erratic or Out of Control as opposed to only 24% of the participants using Dichloromethane having the same flags. As well as the environmental concern in using Freon (or any Chlorofluorocarbon), these results suggest that less consistent performance is obtained when using this solvent for the Oil and Grease test than when using Dichloromethane. This is also visually seen in Figures 2 and 3 in Appendix 1.

Due to the overall low bias noted above, the Automated Evaluation Technique was reapplied to the results, using the interlaboratory median as the centre of reference, rather than the target values. The diagrams are given in Figures 4-6. This results in the centre of the circle representing the area of acceptable performance being shifted away from the target, and includes a different set of participants than in the initial evaluation. (The target value is marked on Figures 4-6.) The Error Lines (Concentration and Intercept) are also shifted, which results in a different value for the PD (Section 3.4, Step 4 v)) for each participant. This may result in a different coding of performance. Both codes of performance are given in Table 4 (Appendix 1), and the review of individual laboratory performance below discusses both performance evaluations.

The improvement in ranking for the several individual participants by using the interlaboratory median as the centre of reference, indicates that there is a group of laboratories that can demonstrate good agreement among themselves. These laboratories also demonstrate good within-laboratory precision. The difference in the interlaboratory median and mean from the target suggests that the Oil and Grease test may not be able to achieve 100% recovery of the motor oil used as the spiking material in this study. This emphasizes the need for reference standards for this procedure to determine the accuracy of this method.

The results for the unspiked effluents (Samples E4 and E5) show a central group of the majority of participants, with several outliers (Figure 7). There were several participants who did not detect any Oil & Grease in these samples. The combination of participants with non-detects and high outliers results in a high standard deviation for these samples (Table 3). There appears more variability among the laboratories using Freon as the extraction solvent than among the laboratories using Dichloromethane, as the participants using Freon have more results that appear erratic (Figure 7). As noted above regarding the need for reference standards, the need for reference matrix materials for this method is emphasized by the variability of results among the laboratories for the effluent samples.

INDIVIDUAL LABORATORY PERFORMANCE

Individual laboratory performance for the spiked reagent water samples is summarized in Table 4, Appendix 1. Performance has been evaluated against the target and against the interlaboratory median, as noted above. An overall assessment has been provided for the performance of the participants for the unspiked effluent samples above.

Laboratories that demonstrated erratic performance, received this flag regardless of whether the target or the interlaboratory median was used as the centre reference point. These laboratories should strive for more consistent application of their analytical procedures.

Only one participant was biased high (Laboratory 9039), regardless which centre

reference point was used. No background laboratory contamination was observed in the unspiked reagent water sample (D4), so it is unclear as to why they achieved over-recovery on the samples in this study.

Laboratories that were flagged low, regardless of whether the interlaboratory median or the target value was used, should investigate their method. Not rinsing the sample bottle with an aliquot of solvent, too high a temperature of the water bath or improper timing of the solvent mixing steps may all contribute to their low bias.

Many of the participants were flagged low relative to the target but were assigned acceptable performance when evaluated against the interlaboratory median. This group of laboratories demonstrate comparable performance within the group and will hopefully continue to maintain this level of performance. It is hoped that when reference standards may come available for this test, this group of laboratories will demonstrate agreement for accuracy as well.

Some of the participants had acceptable performance when evaluated against the target but are assigned high flags when evaluated against the interlaboratory median. These participants are demonstrating greater extraction efficiency with their methods than the main group of laboratories. They should not consider themselves penalized for being higher than the group when evaluated against the interlaboratory median, but should continue to maintain their level of performance.

5 REFERENCES

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5. Cussion, S.; Interlaboratory Study 89-1: Volatile Organic Parameters in Reagent Water and Oil & Grease in Reagent Water and Effluent; 1990; Queen's Printer for Ontario; ISBN 0-7729-7164-1.

6 APPENDIX 1 - TABLES AND GRAPHS

TABLE 1 - OIL & GREASE (MISA TEST GROUP 25)
RESULTS IN mg/L

LAB CODE	D4	D5	D6	E4	E5	EXTRACTION SOLVENT
SPIKE	-	7	22	-	-	
9001	0	5	16	2	5	DICHLOROMETHANE
9004	0.5	0.7	1.4	1.6	2.4	DICHLOROMETHANE
9005	<1 <DL	7	20	6	3	DICHLOROMETHANE
9006	1	6	19	3	3	DICHLOROMETHANE
9007	<1.00	5.00	13.90	<1.00	<1.00	DICHLOROMETHANE
9008A	<DL	9.1	11.3	1.9	1.1	FREON
9008B	<DL	<DL	13.7	2.7	BROKEN	FREON
9009	2.9	7.9	20.4	4.8	6.7	DICHLOROMETHANE
9010	<1	<1	18	<1	5	FREON
9011	<0.1	5.3	17.0	0.1	<0.1	FREON
9012	<0.1	4.3	12.2	<1.0	<1.0	FREON
9013	<0.1	2.0	8.0	0.17	0.28	FREON
9014	<0.4	5.5	18.6	2.5	2.9	DICHLOROMETHANE
9015	14	10.5	10.0	13.0	11.0	DICHLOROMETHANE
9017	<1	6	19	2	2	DICHLOROMETHANE
9018	4.8	6.8	18.7	3.2	4.0	FREON
9019	1.6	7.5	20.4	2.3	2.5	DICHLOROMETHANE
9020	<0.3	6.4	20	3.0	4.1	DICHLOROMETHANE
9021	3	10	21	2	5	FREON
9022	WITHDRAWN FROM STUDY					
9023	BROKEN	BROKEN	18.3	<1.0	<1.0	FREON
9024	<1.0	5.84	22.3	<1.0	2.9	FREON
9025	<1	3	7	4	3	DICHLOROMETHANE
9026	BROKEN	4.6	17	BROKEN	BROKEN	DICHLOROMETHANE
9027	1.3	10.7	20.8	2	3	FREON
9028A	<1	6.0	20.4	2.8	4.0	DICHLOROMETHANE
9028B	<1	6.2	19.4	2.0	3.2	DICHLOROMETHANE
9029	<1	5	15	3	4	HEXANE

LAB CODE	D4	D5	D6	E4	E5	EXTRACTION SOLVENT
SPIKE	-	7	22	-	-	
9030	<0.3	3.7	8.3	1.4	1.8	DICHLOROMETHANE
9030*				1.6		
9031	1.0	8.5	8.2	6.7	8.0	DICHLOROMETHANE
9032	0.20	6.6	BROKEN	2.4	4.2	DICHLOROMETHANE
9033	<1	7	21	4	2	DICHLOROMETHANE
9034	1.2	5.8	17.1	1.5	2.2	FREON
9036	1.4	2.0	20.0	4.3	2.3	DICHLOROMETHANE
9038	4	2	1	8	2	FREON
9039	<0.2	11.0	32.0	0.9	1.2	FREON
9040	<MDL	<MDL	1.3	3.5	3.4	DICHLOROMETHANE
9041	<1.0	9.18	22.31	1.20	2.04	FLUOROCARBON 113
9042	<1	6	19	<1	N/A	FREON
9043	0.2	5.5	17.6	0.5	0.9	PETROLEUM ETHER
9044	0.2	6.6	22.4	0.8	1.0	PETROLEUM ETHER
9045	<10	<10	19.4	<10	<10	FREON
9046	9	12	9	10	24	DICHLOROMETHANE
9047	<5	11	7	<5	<5	FREON
9048	UNABLE TO REPORT RESULTS					

NOTE: Freon = 1,1,2-Trichloro-1,2,2-trifluoroethane
Dichloromethane = Methylene Chloride

* Laboratory 9030 requested a change to their result for sample E4. The revised result was used for the evaluation procedure.

TABLE 2 - OIL & GREASE (MISA TEST GROUP 25)
RESULTS CONVERTED TO PERCENT RECOVERY OF TARGET VALUE
(REAGENT WATER SAMPLES ONLY)

LAB CODE	D5	D6	SOLVENT
SPIKE	7	22	
9001	71%	73%	DICHLOROMETHANE
9004	10%	6%	DICHLOROMETHANE
9005	100%	91%	DICHLOROMETHANE
9006	86%	86%	DICHLOROMETHANE
9007	71%	63%	DICHLOROMETHANE
9009	113%	93%	DICHLOROMETHANE
9008A	130%	51%	FREON
9008B	0%	62%	FREON
9010	0%	82%	FREON
9011	76%	77%	FREON
9012	61%	55%	FREON
9013	29%	36%	FREON
9014	79%	85%	DICHLOROMETHANE
9015	150%	45%	DICHLOROMETHANE
9017	86%	86%	DICHLOROMETHANE
9018	97%	85%	FREON
9019	107%	93%	DICHLOROMETHANE
9020	91%	91%	DICHLOROMETHANE
9021	143%	95%	FREON
9023	BROKEN	83%	FREON
9024	83%	101%	FREON
9025	43%	32%	DICHLOROMETHANE
9026	66%	77%	DICHLOROMETHANE
9027	153%	95%	FREON
9028A	86%	93%	DICHLOROMETHANE
9028B	89%	88%	DICHLOROMETHANE
9029	71%	68%	HEXANE

LAB CODE	D5	D6	SOLVENT
SPIKE	7	22	
9030	53%	38%	DICHLOROMETHANE
9031	121%	37%	DICHLOROMETHANE
9032	94%	BROKEN	DICHLOROMETHANE
9033	100%	95%	DICHLOROMETHANE
9034	83%	78%	FREON
9036	29%	91%	DICHLOROMETHANE
9038	29%	5%	FREON
9039	157%	145%	FREON
9040	0%	6%	DICHLOROMETHANE
9041	131%	101%	FREON
9042	86%	86%	FREON
9043	79%	80%	PETROLEUM ETHER
9044	94%	102%	PETROLEUM ETHER
9045	0%	88%	FREON
9046	171%	41%	DICHLOROMETHANE
9047	157%	32%	FREON

TABLE 3 - STATISTICAL SUMMARY

	ALL PARTICIPANTS		DICHLOROMETHANE ONLY		FREON ONLY		ALL PARTICIPANTS	
SAMPLE	D5	D6	D5	D6	D5	D6	E5	E6
TARGET	7	22	7	22	7	22		
MEAN	5.80	15.93	5.74	14.78	5.82	16.52	1.93	3.33
MEDIAN	6	17.8	6	17.8	6.4	17.55	2	2.7
STD DEV	3.246	6.411	2.826	6.387	3.845	6.966	2.75	4.003
n	37	41	20	21	14	17	42	40
SELECTED MEDIAN	6	18.65	6	19.7	6	18.35		
SELECTED STD DEV	3.132	4.564	2.515	1.930	3.733	0.915		
n	36	36	19	14	13	6		
S_h/S_l	1.457		0.767		0.245		N/A	
S_w - TARGET	0.663		0.594		0.872		N/A	
S_w - MEDIAN	0.397		0.538		0.879		N/A	

TABLE 4 - EVALUATION SUMMARY

	EVALUATED VS TARGET			EVALUATED VS MEDIAN		
LAB CODE	ALL	DICHLOROMETHANE	FREON	ALL	DICHLOROMETHANE	FREON
9001	LS	LS		LS	LS	
9004	LS	LS		LS	LS	
9005	A	WOC		WHI	A	
9006	LS	LS		A	A	
9007	LS	LS		Ls	OC	
9009	AI	AI		HI	WOC	
9008A	ER		ER	ER		ER
9008B	ER		ER	ER		ER
9010	ER		ER	ER		ER
9011	LS		LS	WLS		A
9012	LS		LS	LS		LS
9013	LS		LS	Ls		LS
9014	LS	LS		A	A	
9015	ER	ER		ER	ER	
9017	LS	LS		A	A	
9018	OC		WLS	A		A
9019	AI	AI		HI	A	
9020	WLS	WLS		WHS	A	
9021	OC		OC	He		Hi
9024	A		A	OC		OC
9025	Ls	Ls		Ls	Ls	
9026	Ls	Ls		LI	Ls	
9027	OC		OC	He		Hi
9028A	A	WLI		WOC	A	
9028B	WLS	WLS		A	A	
9029	LS	-	-	LS	-	-
9030	Ls	Ls		Le	Le	
9031	ER	ER		ER	ER	
9033	A	A		HS	A	
9034	LS		LS	WLS		A
9036	Le	Le		ER	OC	
9038	Le		Ls	Le		Ls

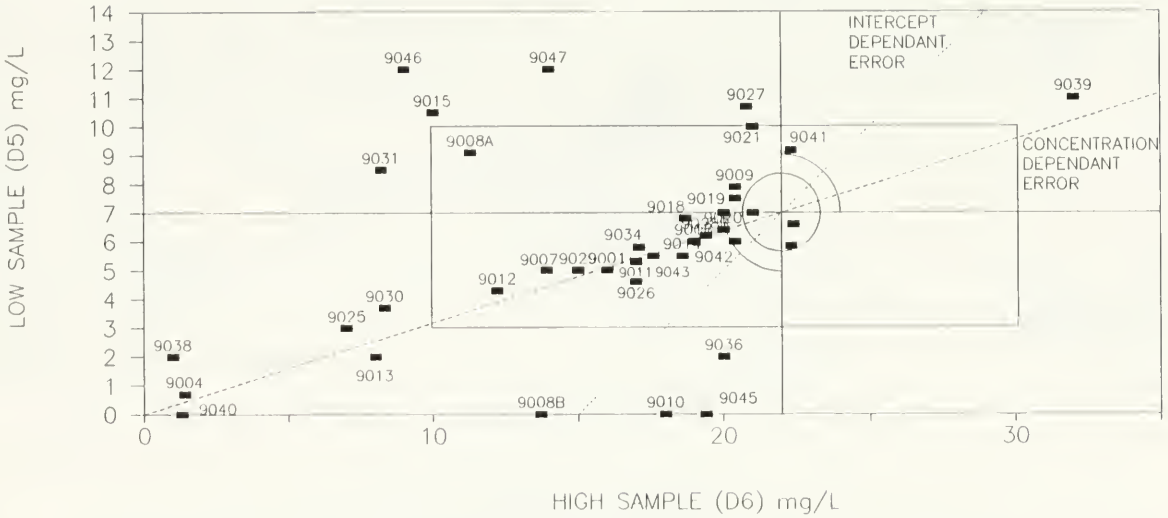
	EVALUATED VS TARGET			EVALUATED VS MEDIAN		
LAB CODE	ALL	DICHLOROMETHANE	FREON	ALL	DICHLOROMETHANE	FREON
9039	Hs		HS	Hs		HS
9040	ER	ER		ER	ER	
9041	WOC		A	HI		HI
9042	LS		WLS	A		A
9043	LS	-	-	A	-	-
9044	A	-	-	HS	-	-
9045	ER		ER	ER		ER
9046	ER	ER		ER	ER	
9047	ER		ER	ER		ER

KEY TO SUMMARY TABLE

A	Acceptable Performance
WAI	Warning: Slight imprecision
WLI	Warning: Biased low, Probable intercept problem
WHI	Warning: Biased high, Probable intercept problem
WLS	Warning: Biased low, Probable slope problem
WHS	Warning: Biased high, Probable slope problem
WOC	Warning: Out of control - one result erratic
LI	Biased Low, Probable intercept problem
LS	Biased low, Probable slope problem
Li	Biased low, Possible intercept problem
Ls	Biased low, Possible slope problem
L	Biased low
Le	Biased low and/or erratic
HI	Biased high, Probable intercept problem
HS	Biased high, Probable slope problem
Hi	Biased high, Possible intercept problem
Hs	Biased high, Possible slope problem
H	Biased high
He	Biased high and/or erratic
OC	Out of control - one result erratic
ER	Both results erratic

FIGURE 1: INTERLABORATORY STUDY 91-4

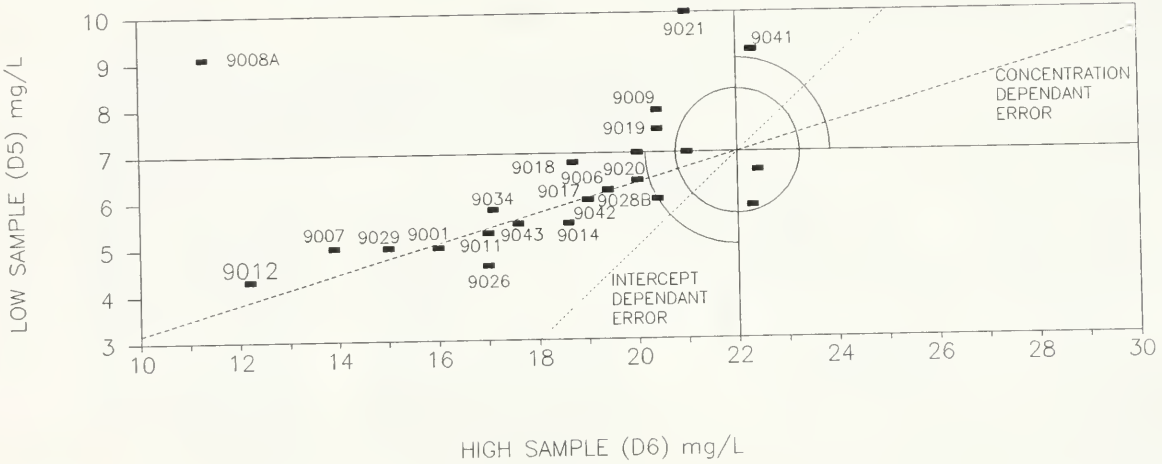
OIL & GREASE



AREA INSIDE BOX EXPANDED IN FIGURE 1A.

FIGURE 1A: INTERLABORATORY STUDY 91-4

OIL & GREASE



EXPANDED AREA FROM FIGURE 1.

FIGURE 2: INTERLABORATORY STUDY 91-4

OIL & GREASE - SOLVENT: DICHLOROMETHANE

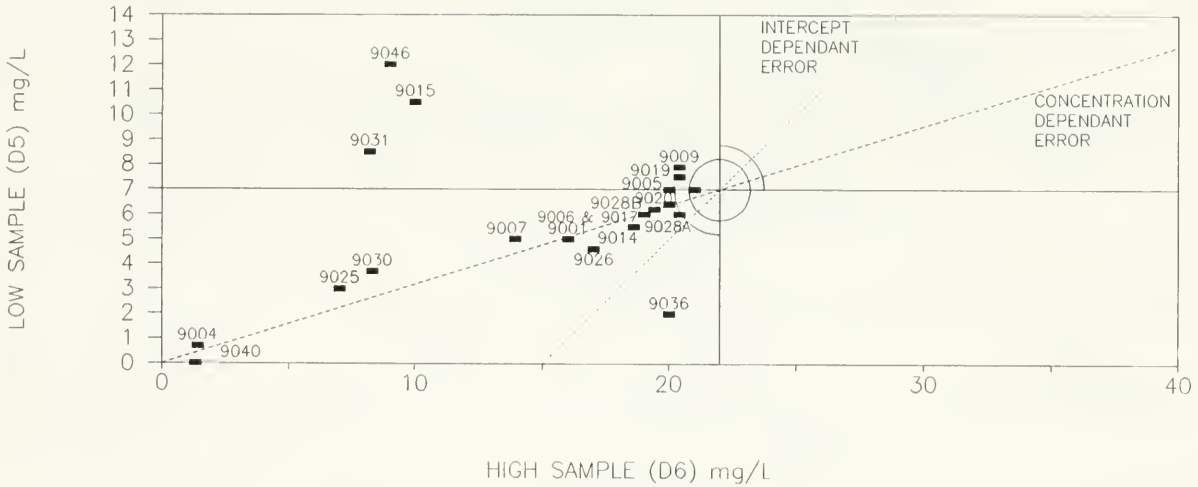


FIGURE 3: INTERLABORATORY STUDY 91-4

OIL & GREASE – SOLVENT: FREON

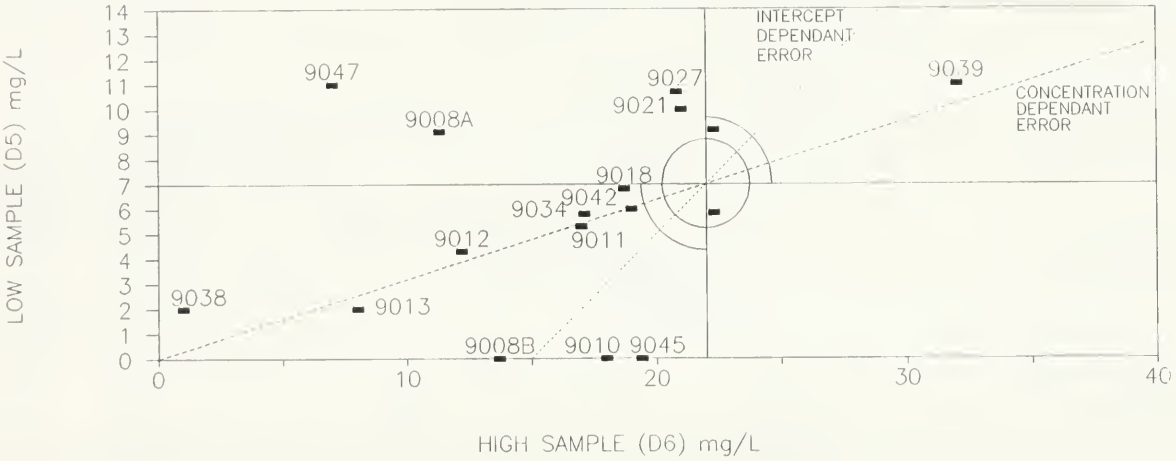
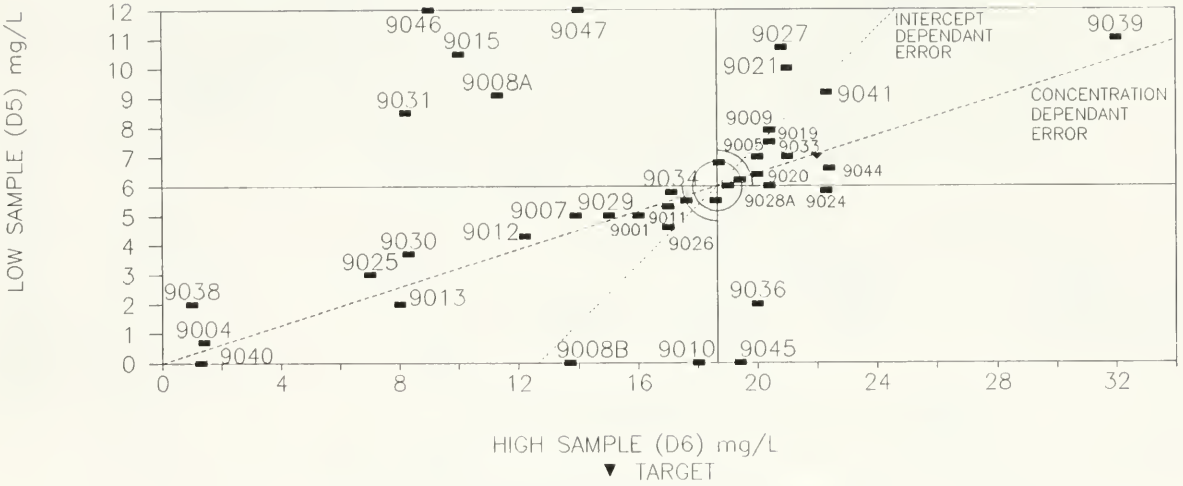


FIGURE 4: INTERLABORATORY STUDY 91-4

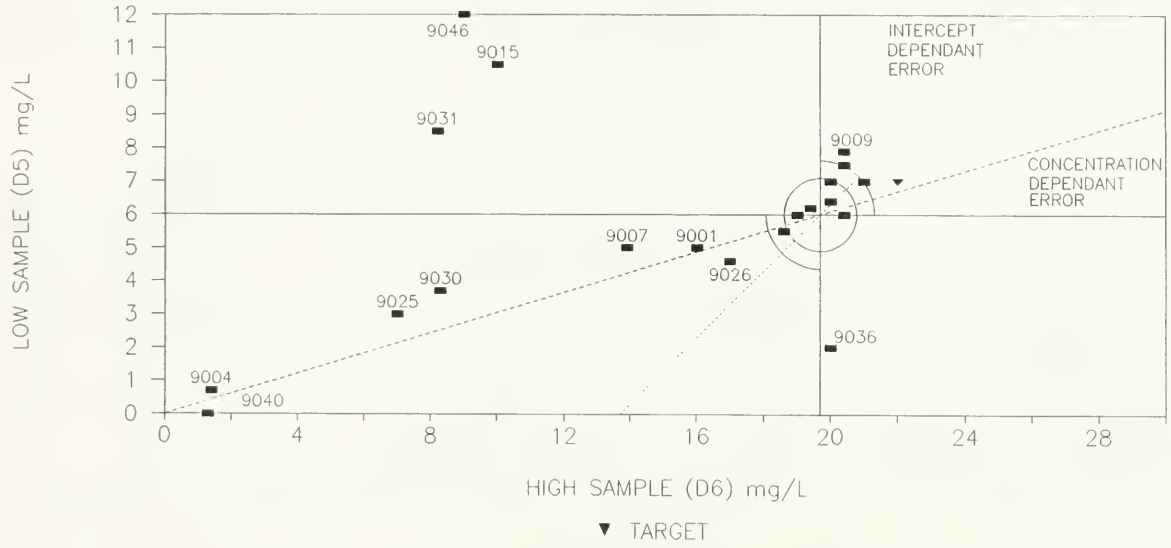
OIL & GREASE



RESULTS EVALUATED AGAINST INTERLABORATORY MEDIAN

FIGURE 5: INTERLABORATORY STUDY 91-4

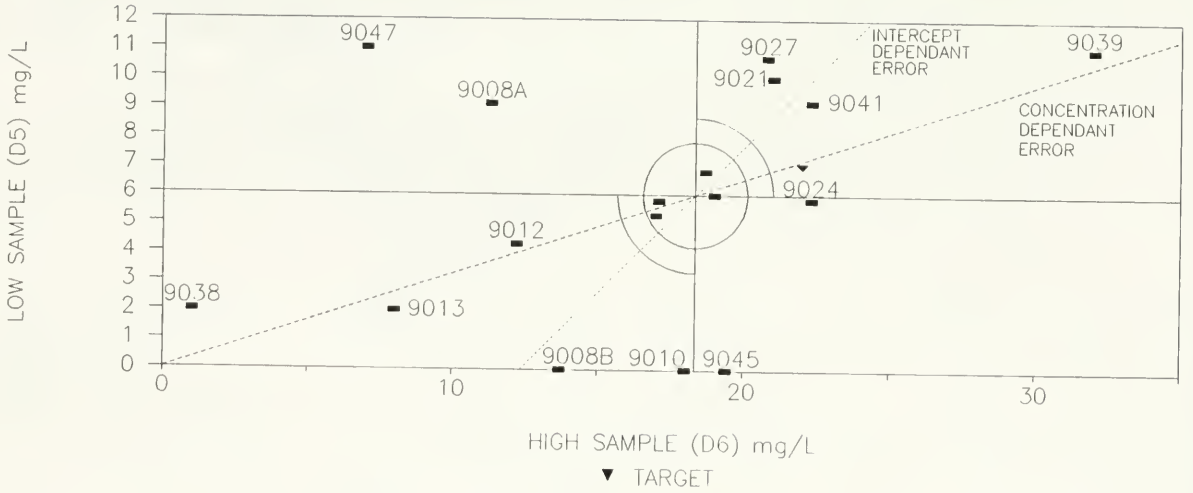
OIL & GREASE – SOLVENT: DICHLOROMETHANE



RESULTS EVALUATED AGAINST INTERLABORATORY MEDIAN

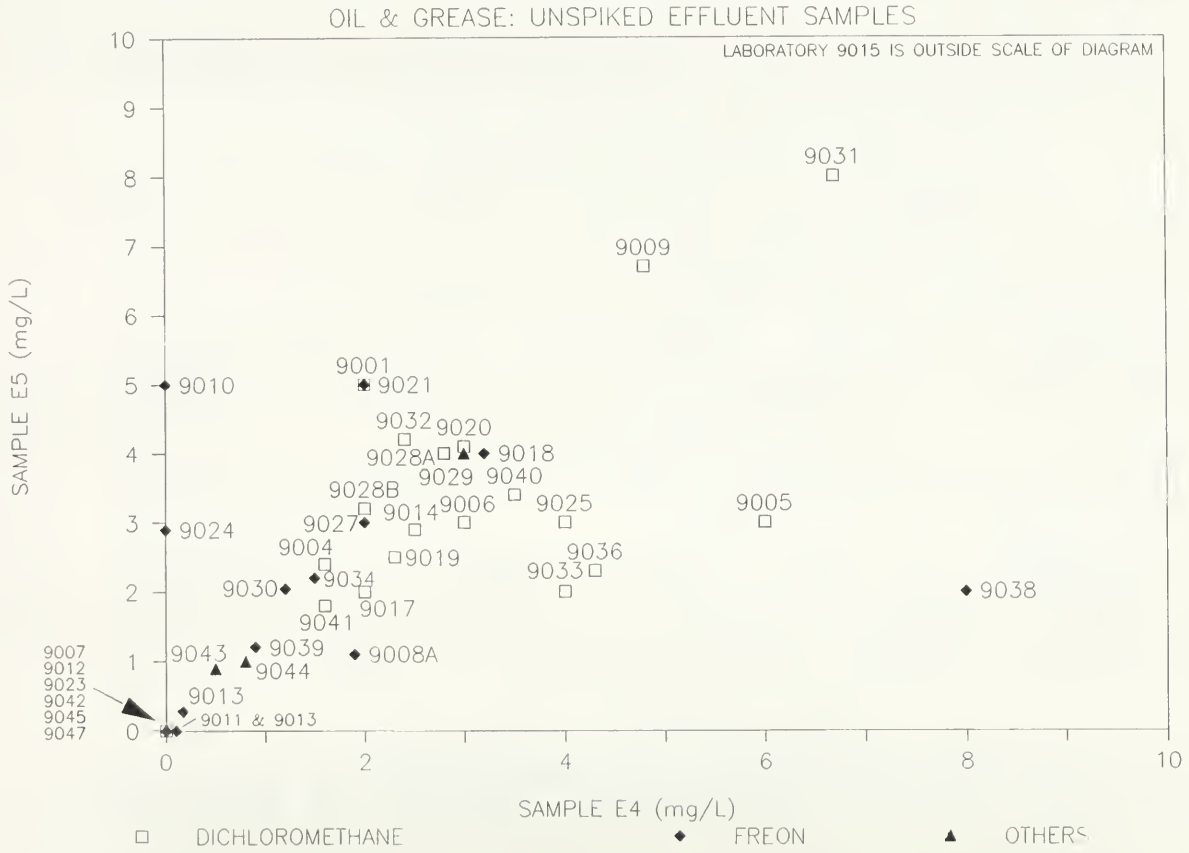
FIGURE 6: INTERLABORATORY STUDY 91-4

OIL & GREASE - SOLVENT: FREON



RESULTS EVALUATED AGAINST INTERLABORATORY MEDIAN

FIGURE 7: INTERLABORATORY STUDY 91-4



7 APPENDIX 2 - LIST OF PARTICIPANTS AND CORRESPONDENCE

LIST OF PARTICIPANTS

Ontario Ministry of the Environment
Laboratory Services Branch
Inorganic Trace Contaminants Section
Hazardous Waste Unit
125 Resources Rd.
Rexdale, Ontario, M9W 5L1
Contact: Janet Mills/Ann Jones
235-6075/2074

Barringer Laboratories Ltd.
5735 McAdam Rd.
Mississauga, Ontario
L4Z 1N9

Contact: Dr. Michael Dancziger
890-8566

Placer Dome Inc.
Dome Mine
P.O. Box 70
South Porcupine, Ontario
P0N 1H0

Contact: Ron Millions
(705) 235-3221

Accurassay Labs. Ltd.
P.O. 426
3 Industrial Dr.
Kirkland Lake, Ontario
P2N 3J1

Contact: Dr. George Duncan
(705) 567-3361

Bondar Clegg
5420 Canotek Rd.
Ottawa, Ontario
K1J 8X5

Contact: Mike Ziebell
(613) 749-2220

EAG Analytical Services
475 Cochrane Dr., Unit #13
Markham, Ontario
L3R 9R5

Contact: Suman Punani
479-6107

BAS Labs Ltd.
14 Abacus Road
Brampton, Ontario
L6T 5B7

Contact: Daniel Andrews
458-4505

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66 Mohawk St., Unit 19
Brantford, Ontario
N3S 2W3

Contact: Helen Graham
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86 Rankin Street
Waterloo, Ontario
N2V 1V9

Contact: Wayne Smith
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Shell Canada Products Ltd.
Sarnia Manufacturing Centre
Refinery Laboratory
Corunna, Ontario
N0N 1G0

Contact: Dave Pillon
(519) 481-1396

Novalab
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Lachine, Quebec
H8T 1A1

Contact: Dr. Pierre Bedard
(514) 636-6218

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London, Ontario
N6N 1B2

Contact: Debbie Boersma
(519) 668-1005

Esso Petroleum Canada
Nanticoke Refinery
Jarvis, Ontario
N0A 1J0

Contact: Peter Vaughan/Geoff Golding
(519) 587-7011

Ontario Hydro
Chemical Research Dept.
800 Kipling Ave., KR310
Toronto, Ontario
M8Z 5S4

Contact: Dr. Otto Herrmann
231-4111 Ext. 6262

CanTest Ltd.
Suite 200
1523 West 3rd Ave.
Vancouver, B.C.
V6J 1J8

Contact: Robert Hunter
(604) 734-7276

Inco Ltd.
Central Process Technology
Copper Cliff, Ontario
P0M 1N0

Contact: Dave Maskery
(705) 682-5563

Paracel Laboratories Ltd.
2319 St. Laurent Blvd., Unit #100
Ottawa, Ontario
K1N 7N3

Contact: Dr. William G. Craig
(613) 731-9577

Walker Laboratories
2800 Townline Rd.
P.O. Box 100
Thorold, Ontario
L2V 3Y8

Contact: Brian Fell
(416) 227-1158, ext 279

Proctor and Redfern
45 Greenbelt Drive
Don Mills, Ontario
M3C 3K3

Contact: Denise Archer
445-3600

Pollutech Ltd.
768 Westgate Rd.
Oakville, Ontario
L6L 5N2

Contact: Erin Teeft
847-0065

XRAL Environmental
1903 Leslie Street
Don Mills, Ontario
M3B 2M3

Contact: Karen Lopez
445-5809

Novacor Chemicals (Canada) Ltd.
785 Hill St.
Corunna, Ontario
N0N 1G0

Contact: Percy Holder
(519)862-2911 ext. 2268

Zenon Environmental Inc.
8577 Commerce Ct.
Burnaby, B.C.
V5A 4N5

Contact: Dr. Dorothy Jeffery
(604) 444-4808

Canviro Analytical Laboratories Ltd.
50 Bathurst Dr., Unit 12
Waterloo, Ontario
N2V 2C5

Contact: Annette Bibaud
(519) 747-2575

Enviroclean
921 Leathorne Street
London, Ontario
N5Z 3M7

Contact: Lynn Arsenault
(519) 686-7558

Technical Service Laboratories
1301 Fewster Dr.
Mississauga, Ontario
L4W 1A2

Contact: Nick Boulton
625-1544

Lakefield Research
185 Concession St.
Lakefield, Ontario
K0L 2H0

Contact: David Hevenor
(705) 652-3341

Atomic Energy of Canada Ltd.
Chalk River Nuclear Laboratories
Chalk River, Ontario
K0J 1J0

Contact: Keith R. Betty
(613) 584-3311 ext. 3173

Williams Operating Corp.
Williams Minesite
P.O. Bag 500
Marathon, Ontario
P0T 2E0

Contact: Victor Rafuse
(807) 238-1100 ext. 226

Sunoco - Sarnia Refinery
Box 307
Sarnia, Ontario
N7T 7J3

Contact: Dave MacMillan/Don Waugh
(519) 337-2301 ext. 417/227

Ontario Ministry of the Environment
Laboratory Services Branch
Trace Organics Section
Special Organics Unit
125 Resources Rd., Box 213
Rexdale, Ontario, M9W 5L
Contact: Paul Yang/Maryann Bogard
235-5754/5932

Environmental Protection Laboratories Inc.
6850 Goreway Dr.
Mississauga, Ontario
L4V 1P1

Contact: Luc Dionne/Tim Munshaw
673-3255

Clayton Environmental Consultants
949 McDougall St.
Windsor, Ontario
N9A 1L9

Contact: Dr. James P. Johnson
(519) 255-9797

Petro Canada Ltd.
Lake Ontario Refinery
Oakville Plant
Mississauga Street North
Oakville, Ontario
L6J 5B5

Contact: Charlie Withnell
825-1757

Manitoba Environment
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745 Logan Ave.
Winnipeg, Manitoba
R3E 3L5

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(204) 945-1156

Wastewater Technology Centre
867 Lakeshore Rd.
Box 5068
Burlington, Ontario
L7R 4L7

Contact: Jim Fraser
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Zenon Environmental Inc.
5555 North Service Rd.
Burlington, Ontario
L7L 5H7

Contact: Mary Pejic
(416) 332-8788

B.C. Research
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Vancouver, B.C.
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Petro Canada Ltd.
Lake Ontario Refinery
Mississauga Plant
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Mississauga, Ontario
L5J 2Y3

Contact: Peter Hay
849-5246

Near North Laboratories
191 Booth Rd., Unit 11
R.R.#5
North Bay, Ontario
P1B 8Z4

Contact: Brenda McLay
(705) 497-0550

Regional Municipality of Waterloo
Engineering Laboratory
100 Maple Grove Rd.
R.R. #31
Cambridge, Ontario
N3H 4R6

Contact: David Andrews
(519) 885-9500

ASL Analytical Service Laboratories Ltd.
1988 Triumph St.
Vancouver, B.C.
Y5L 1K5

Contact: James R.Downie
(604) 253-4188

MOE INTERLABORATORY VARIABILITY STUDY NOTIFICATION FOR THE ANALYSIS OF TRACE INORGANIC COMPOUNDS

STUDY NO. 91-3, STUDY 91-4, & STUDY 91-5

INTRODUCTION

Laboratories receiving this notification are invited by Environment Ontario, Laboratory Services Branch, to participate in three different interlaboratory variability studies of spiked reagent water and spiked mixed effluent, conducted using MISA analysis protocols. Laboratories interested in participating in this program, scheduled for three successive weeks commencing with the week of October 21, 1991, should contact Sylvia Cussion at (416) 235-5842 of Environment Ontario to confirm participation. Written confirmation (attached response sheet) must be submitted to Environment Ontario no later than October 15, 1991 (FAX: (416) 235-6110).

BACKGROUND

This study is being conducted to assist laboratories in assessing their analytical performance in conjunction with the MISA program. All procedures should follow those principles and protocols outlined in the MISA regulations (Ontario Reg. 695/88 as amended to Ontario Reg. 533/89). These studies target three different MISA test groups. Separate sets of samples will be distributed for each analytical test, one set each per week, for three successive weeks. Sample sets will include spiked reagent water samples and effluent matrix samples. Laboratories may choose to participate in only one, two, or all three of the studies.

The sample schedule is as follows:

STUDY	TEST	MISA Group	No. of Samples	Matrix	Preservative	Study Date
91-3	Phenolics	14	3	Reagent Water	H_2SO_4	October 22, 1991
	(4AAP)		3	Effluent	H_2SO_4	October 22, 1991
91-4	Solvent	25	3	Reagent Water	None	October 29, 1991
	Extractables		2	Effluent	None	October 29, 1991
91-5	Organic Carbon	5a & b	3	Reagent Water	H_2SO_4	November 4, 1991
	DOC & TOC		3	Effluent	H_2SO_4	November 4, 1991

NOTE: If any of the above preservatives present an analytical problem, please indicate this on the response form.

Participating laboratories are expected to analyze the samples within the time limits specified in Schedule 2 of the general MISA regulations (Ontario Reg. 695/88). Blank reporting forms will be provided with the samples. Results for all analyses are to be reported within thirty (30) days of receipt of the samples to Sylvia Cussion at the following address:

Environment Ontario
Laboratory Services Branch
Quality Management Unit
125 Resources Rd., P.O. Box 213
Rexdale, Ontario
M9W 5L1

SUMMARY OF RESULTS

All participating laboratories will be assigned a unique identification code. All laboratories reporting results will receive a complete set of the results where they will be identified only by the identification code. Recommendations made by Environment Ontario will be included in the final report. Results will remain confidential and will only be released with the written permission of the individual participants.

It is the intent of this interlaboratory study (along with others) to assess the interlaboratory variability and detection capability for a broad range of organics and inorganics.

MOE INTERLABORATORY VARIABILITY STUDY NOTIFICATION FOR THE ANALYSIS OF MERCURY

STUDY 91-3, STUDY 91-4 & STUDY 91-5

We will participate for the following study(ies):

	YES	NO	Minimum Sample Volume Required
Study 91-3 Phenolics (4AAP)	☐	☐	_____
Study 91-4 Solvent Extractables	☐	☐	_____
Study 91-5 Organic Carbon (DOC & TOC)			_____

For the completeness of our records, and to avoid any shipping delays, please fill in the following:

Mailing Address:

_____

_____

_____

_____

Shipping Address:

_____

_____

_____

_____

Contact Person: _____

Telephone: _____

Please return this response form by October 15, 1991 to:

Sylvia Cussion
Environment Ontario
Laboratory Services Branch
Quality Management Unit
125 Resources Rd., P.O. Box 213
Rexdale, Ontario, M9W 5L1

FAX: (416) 235-6110

Quality Management Unit
(416) 235-5842

October 29, 1991

TO: PARTICIPANTS OF INTERLABORATORY STUDY 91-4

Please find enclosed five 1000 mL glass bottles for the analysis of Solvent Extractables (Oil and Grease), MISA Test Group 25. The samples are not preserved. The samples are labelled as follows:

Distilled water:	D4	D5	D6
Effluent:	E4	E5	

If you are missing any of the above items, please contact me at the above phone number immediately.

As stated in the notification distributed September 27, 1991, samples should be analyzed using the principles and protocols outlined in the MISA General Regulation (Ontario Reg. 695/88, as amended to Ont. Reg. 533/89). Store all samples in a refrigerator at 4 degrees Celcius until ready for analysis. Time limit for storage is 7 days.

To ensure timely release of a summary report, results are to be submitted by December 6, 1991. Report forms to be used are included with the samples. Please identify all sample results with your lab identification number and the sample numbers described above. Please contact me if there are any problems or questions re the interlaboratory study. Thank you for your participation.

Your lab identification number is:

Sincerely,

Sylvia Cussion
Lab Quality Audit Scientist
(416) 235-5842

Quality Management Unit
(416) 235-5842

October 29, 1991

TO: PARTICIPANTS OF INTERLABORATORY STUDY 91-4

Please find enclosed ten 1000 mL glass bottles for the analysis of Solvent Extractables (Oil and Grease), MISA Test Group 25. The samples are not preserved. The samples (two of each) are labelled as follows:

Distilled water:	D4	D5	D6
Effluent:	E4	E5	

If you are missing any of the above items, please contact me at the above phone number immediately.

As stated in the notification distributed September 27, 1991, samples should be analyzed using the principles and protocols outlined in the MISA General Regulation (Ontario Reg. 695/88, as amended to Ont. Reg. 533/89). Store all samples in a refrigerator at 4 degrees Celcius until ready for analysis. Time limit for storage is 7 days.

To ensure timely release of a summary report, results are to be submitted by December 6, 1991. Report forms to be used are included with the samples. Please identify all sample results with your lab identification number and the sample numbers described above. Please contact me if there are any problems or questions re the interlaboratory study. Thank you for your participation.

Your lab identification number is:

Sincerely,

Sylvia Cussion
Lab Quality Audit Scientist
(416) 235-5842

INTERLABORATORY STUDY 91-4

SOLVENT EXTRACTABLES (OIL & GREASE)

REPORT FORM

OCTOBER 29, 1991

REPORT DUE DATE: DECEMBER 6, 1991

LAB IDENTIFICATION CODE:

SAMPLE	RESULT (mg/L)
D4	
D5	
D6	
E4	
E5	

SAMPLE PREPARATION PRINCIPLES:

SOLVENT USED FOR EXTRACTION:

INSTRUMENTAL MEASUREMENT METHOD PRINCIPLES:

Quality Management Unit
(416) 235-5842

January 7, 1992

TO: PARTICIPANTS OF INTERLABORATORY STUDY 91-4

Thank you for your participation in Interlaboratory Study 91-4 for the analysis of Solvent Extractables (Oil & Grease). Please find attached a copy of the table of results. Please note that some participants analyzed the samples in duplicate. The results have been reported separately as "A" or "B". If there are any transcription errors, please contact me no later than January 17, 1992. A report evaluating the results will be prepared and a copy provided to all participants.

Your lab identification number is:

Sincerely,

Sylvia Cussion
Lab Quality Audit Scientist
(416) 235-5842

ATTACHMENT